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The Calcium and Magnesium Content of **RAW COTTON FIBER**

A Progress Report

Agricultural Research Service

U. S. DEPARTMENT OF AGRICULTURE

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THE CALCIUM AND MAGNESIUM CONTENT OF RAW COTTON FIBER

A Progress Report

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ABSTRACT

This is a progress report on a continuing investigation concerned with the calcium and magnesium content of raw cotton fiber. Analyses have been made in an attempt to determine whether the fiber's content of these elements might be related to its area or year of production, its grade, to fertilizer practices, to the extent of preharvest weathering, or to other factors. The results are presented in parts per million in the fiber of calcium, magnesium, and "total calcium", the latter representing the two elements expressed together as calcium.

The following tentative conclusions have been derived from the data:

(1) Within the United States Cotton Belt there was a tendency for high-grade Western and Texas-Oklahoma cotton to be somewhat higher on the average in "total calcium", calcium, and calcium-magnesium ratio than Midsouthern and Southeastern cotton, but the differences were not very large nor very regular.

(2) Preliminary analyses disclosed a definite relationship between the calcium, "total calcium", and calcium-magnesium ratio on the one hand and the grade of the fiber on the other, the lower grades being characterized by higher levels in each of these three properties than the higher grades. Subsequent data showed, however, that this relationship represented an artifact resulting from the circumstance that the lower grades of cotton contained more leaf trash than the higher ones. The leaf material, which had not been removed from the fiber in the preliminary analyses, was very much higher in calcium content than the fiber itself, likewise higher in calcium-magnesium ratio.

(3) A suggestion has come from commercial sources that cotton of recent production might be higher in calcium and magnesium than cotton produced several years ago. Analyses to date do not support this idea.

(4) Fiber from plants suffering from extreme potassium deficiency exhibited a somewhat higher level of calcium and of calcium-magnesium ratio than fiber from similar plants adequately supplied with potassium. Wide variations in the amounts of phosphorus and nitrogen applied did not materially affect the levels of calcium or magnesium in the fiber.

(5) Analyses of fiber subjected to rainfall during a 10-week preharvest weathering period disclosed somewhat variable but generally low levels of loss

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of calcium and magnesium from the fiber. Calcium was more retentive on the fiber than magnesium.

(6) Limited data relating to the degree of variation in calcium and magnesium content of fiber from one boll to the next on a single plant have disclosed a rather low degree of variability. A greenhouse-grown plant analyzed in this fashion showed a consistently higher calcium-magnesium ratio than a field-grown plant.

BACKGROUND

Additional information is urgently needed in respect to quality factors in raw cotton fiber. With increasing frequency in recent years this opinion has been heard from all segments of the cotton industry. Many physical and chemical properties of the fiber need to be measured, the degree and causes of variation in such properties determined, and the various factors related to the performance of the fiber in commercial situations.

Little information is to be found in the literature on variations in the mineral content of raw cotton fiber in relation to the area of growth or to production practices. In 1959, a paper² was published from the authors' laboratory in which iron contamination was shown to be characteristic of certain types of brown spots in raw cotton, spotted cotton of this nature originating particularly in the southeastern part of the U.S. Cotton Belt. With the exception of this paper, however, there seems to be no further information in the literature on this general subject.

EXPERIMENTAL RESULTS

Relation to areas of growth

Numerous raw cotton samples from various parts of the U.S. Cotton Belt, including fibers from experimental plots and commercial sources, have been analyzed for calcium and magnesium. The samples shown in Table 1 were grown in experimental plots at 13 locations for 3 years. In these plots there were two definite deviations from common commercial cultural practices, namely, (1) the fiber was picked promptly after the bolls had opened, and (2) the variety which was used, Coker 100 W, is not the predominant cotton variety in all of the noted locations. Examination of the regional averages in Table 1 reveals a trend for higher "total calcium" and calcium in the Texas-Oklahoma and Western areas as compared with the same for the Southeast and Midsouth, also a higher calcium-magnesium ratio.

In further examination of the matter of calcium and magnesium content in relation to area of growth, analyses were carried out for "total calcium" on a series of Middling commercial samples from the 1958 crop. The data (Table 2) again showed a trend for Texas-Oklahoma and Western cotton to be somewhat

²Harrison, G. K., and P. B. Marsh. 1959. Detection and geographical origin of iron-spotted raw cotton. Amer. Dye-stuff Repr., 48:47-50.

TABLE 1.--Parts per million of "total calcium," calcium, and magnesium in cotton fiber of the variety Coker 100 W; samples free of leaf trash. The average Ca/Mg ratio for Texas-Oklahoma and Western = $890/702 = 1.27$, for the Southeast and Mid-South = $639/658 = .97$. The average "Total Ca" for Texas-Oklahoma and Western/Southeast and Midsouth = $2032/1744 = 1.16$

Location of growth	"Total calcium"			Calcium			Magnesium		
	1947	1948	1949	1947	1948	1949	1947	1948	1949
Texas-Oklahoma and Western									
Areas:									
Chickasha, Okla.....	3000	2000	2200	1650	650	1050	820	820	700
College Station, Tex..	1925	1725	1730	925	700	800	610	620	540
Greenville, Tex.....	2275	1625	2510	1150	675	1350	685	775	700
Sacaton, Ariz.....	2175	2140	1900	925	1000	875	760	680	620
Shafter, Calif.....	1600	1650	2000	575	575	875	620	650	680
State College, N. Mex.	2100	1975	2050	825	725	700	775	760	820
Average.....	2179	1852	2065	1008	721	942	712	718	677
Southeast and Midsouth									
Areas:									
Auburn, Ala.....	1800	1850	2000	800	825	500	610	335	910
Florence, S. C.....	1550	1625	1450	500	500	450	640	680	610
Jackson, Tenn.....	1850	2000	1530	850	675	650	610	800	530
St. Joseph, La.....	2000	1700	1930	775	600	650	745	670	775
Statesville, N. C.....	1700	1950	1720	625	675	550	650	775	715
Stoneville, Miss.....	2075	1675	1700	700	625	750	835	640	580
Tifton, Ga.....	1825	1350	1350	725	500	500	670	520	520
Average.....	1828	1736	1668	711	628	578	680	631	663

higher in "total calcium" than Midsouth and Southeast cotton. The differences in regional averages, however, are not very great (Table 2) and the results for the individual analyses exhibit considerable overlapping among the different areas. The ratio of "total calcium" for Texas-Oklahoma and Western to the same for the Southeast and Midsouth = $1693/1503 = 1.13$, this ratio being quite close to that of 1.16 in the data of Table 1.

Relation to grade

In order to investigate any possible relationship of calcium or magnesium content of commercial raw cotton to the fiber's grade, a series of samples representing four grades and four areas of U. S. production were analyzed. The results (Table 3) show a definite and quite regular increase in "total calcium" and calcium as the grades become lower, this being particularly true in the Western, Texas-Oklahoma and Midsouthern areas, also a higher Ca/Mg ratio in general in the lower-grade samples.

TABLE 2.--Parts per million of "total calcium" in Middling cotton (1958) from several areas. (Each figure represents a bale.)

Location	Sample 1	Sample 2	Sample 3	Sample 4	Average for area
Southeastern Area:					
Atlanta, Ga.....	1755	1925	1500	1700	
Augusta, Ga.....	1500	1650	1600	1750	
Birmingham, Ala.....	1650	1550	1450	1450	
Columbia, S. C.....	1575	1650	1500	1425	
Montgomery, Ala.....	1600	1500	1375	1475	
Raleigh, N. C.....	1500	1825	1700	1450	
					1585
Midsouth Area:					
Alexandria, Miss.....	1375	1350	1150	1225	
Blytheville, Ark.....	1550	1525	1450	1325	
Greenwood, Miss.....	1300	1450	1200	1350	
Hayti, Mo.....	1525	1575	1450	1350	
Little Rock, Ark.....	1400	1425	1700	1525	
Memphis, Tenn.....	1650	1550	1800	1350	
Winnsboro, La.....	1350	1200	1225	1500	
					1422
Texas-Oklahoma Area:					
Abilene, Tex.....	1475	1575	1950	2100	
Altus, Okla.....	1750	1675	1575	1750	
Austin, Tex.....	1525	1325	1375	1775	
Dallas, Tex.....	1225	1375	1700	1900	
Lamesa, Tex.....	1650	1750	1525	1600	
Lubbock, Tex.....	1775	1650	1750	1850	
Munday, Tex.....	1700	1475	1750	1875	
Oklahoma City, Okla.....	1700	1600	1575	1525	
					1650
Western Area:					
Bakersfield, Calif.....	1550	1750	2000	1500	
Carlsbad, N. Mex.....	1500	1600	1750	1625	
El Centro, Calif.....	1400	1825	1800	2700	
El Paso, Tex.....	1700	1850	1800	1750	
Fresno, Calif.....	1900	1925	1600	1825	
Pecos, Tex.....	1775	1650	1500	1550	
Phoenix, Ariz.....	1800	1625	1850	1550	
					1738

The commercial samples analyzed in the experiment of Table 3 contained variable amounts of leaf fragments. It was considered as a possibility that the relationships between grade and calcium content observed in these samples might be concerned with a variation in the amount of contamination of the fiber with calcium from leaf material. Consequently, four samples of Low Middling cotton were analyzed before and after cleaning the leaf fragments out of the fiber. The data (Table 4) indicate that leaf fragments are, in fact, a major source of calcium. The differences in calcium and "total calcium" associated with cleaning (Table 4) seemed of sufficient magnitude to explain the relationship of mineral content to grade observed in Table 3. In further examination of this same question, analyses were carried out on trash from commercial cotton obtained in a Shirley Analyzer.

TABLE 3.--Relationship of parts per million of "total calcium," calcium, and magnesium to grade and location of growth in commercial cotton samples from the crop of 1959. (Note higher values of "total calcium", calcium, and calcium/magnesium ratio in the lower grades.)

Grade	Western	Texas- Oklahoma	Midsouth	South- eastern
"Total calcium"				
Strict Middling.....	1653*	1483	1544	1458
Middling.....	1703	1547	1572	1492
Strict Low Middling.....	1914	1800	1592	1533
Low Middling.....	2308	1825	1880	1517
Calcium				
Strict Middling.....	661	592	544	500
Middling.....	714	703	544	550
Strict Low Middling.....	964	869	589	561
Low Middling.....	1167	939	828	589
Magnesium				
Strict Middling.....	603	539	602	581
Middling.....	603	514	611	575
Strict Low Middling.....	578	563	600	583
Low Middling.....	692	533	642	561
Ca/Mg ratio				
Strict Middling.....	1.10	1.10	0.90	0.86
Middling.....	1.18	1.37	.89	.96
Strict Low Middling.....	1.67	1.54	.98	.96
Low Middling.....	1.69	1.76	1.29	1.04

*Each figure in this table represents an average of single determinations on classers' samples from nine individual bales.

TABLE 4.--Calcium and magnesium content of Low Middling classers' samples, crop of 1959, before and after hand cleaning to remove leaf fragments.

Sample	"Total calcium", p.p.m.	Ca, p.p.m.	Mg, p.p.m.	Ca/Mg ratio
No. 77, not cleaned.....	2250	1150	675	1.70
No. 77, cleaned.....	1850	935	600	1.56
No. 84, not cleaned.....	2600	1850	450	4.11
No. 84, cleaned.....	1900	1000	550	1.82
No. 85, not cleaned.....	2700	1400	800	1.75
No. 85, cleaned.....	2200	1100	650	1.69
No. 86, not cleaned.....	2900	1300	975	1.33
No. 86, cleaned.....	2100	1000	650	1.54

The data (Table 5) show extremely high "total calcium" and calcium in the trash, in the magnitude of 25 or more times as high as in the lint. Although a difference in the level of calcium and magnesium between leaf and fiber was not unexpected, the magnitude of the difference actually obtained had not been anticipated. It seems highly probable from the data of Tables 4 and 5 that the differences in "total calcium" and calcium associated with grade in Table 3 were in fact related to the amount of leaf trash in the samples.

Relation to year of production

Individuals from certain concerns engaged in cotton dyeing have suggested that the content of calcium and magnesium in cotton of recent growth may be higher than was the case with cotton produced several years ago. The results shown in Table 6, when compared with the data of Table 2, appear to indicate that the content of these elements in recent commercial cotton is actually probably of the same order of magnitude as it has been for some years.

TABLE 5.--"Total calcium", calcium, and magnesium content of trash from commercial cotton obtained in a Shirley Analyzer.

Sample	"Total calcium", p.p.m.	Ca, p.p.m.	Mg, p.p.m.	Ca/Mg ratio
Austin, Tex. Strict Low Middling.....	46000	34000	8000	4.25
Corpus Christi, Tex. Low Middling.....	48000	37000	7000	5.29
El Centro, Calif. Middling.....	49000	33000	9000	3.68

TABLE 6.--"Total calcium" content and percent ash of old samples of cotton.

Year of Growth	Source	"Total calcium", p.p.m.	Percent ash
1862.....	ACCO ¹	1450	1.20
1890.....	AMS ²	1425	--
1899.....	ACCO	2150	1.49
1931.....	ACCO	1850	1.11
1937.....	ACCO	1600	1.19
1937.....	ACCO	1500	1.01
1940.....	ACCO	2250	1.55

¹ Anderson, Clayton, & Company, samples sent to Beltsville, July 1960.

² Agricultural Marketing Service, samples sent to Beltsville, July 1960.

Relation to production practices

Defoliation

Some individuals concerned with the dyeing of cotton have expressed the thought that the calcium content of commercial cotton may have been increased in recent years by the application of calcium-containing defoliants. The matter is one of interest to dyers because of the known effect of calcium on the shade of certain dyes. While it is true that one of the defoliants that has been widely used, calcium cyanamid, is of this nature, the total amount of calcium applied per acre is small. Furthermore, analyses show, Tables 1 and 2, that the "total calcium" and calcium content of the fiber from Southeastern and Midsouthern areas of production, where this defoliant has been much used, are in fact somewhat lower than is the case for fiber from those irrigated and dry areas where it is not used.

Fertilizer practice

An attempt was made to determine whether the calcium or magnesium content of cotton fiber may be related to the amounts of nitrogen, phosphorus, and potassium supplied to the plant. At the Pee Dee Experiment Station at Florence, S.C., certain experimental fertility plots referred to as the "Keitt plots" have been maintained under constant fertilization regimes for over 40 years. The cotton on these plots is grown with varying levels of nitrogen, phosphorus, and potassium. Would the amounts of any of these fertilizer applications influence the level of calcium or magnesium in the fiber? Data relating to this question are shown in Table 7. It may be noted that variations in the amount of nitrogen applied in the fertilizer did not materially influence the percent ash, "total calcium," calcium, or magnesium. The same was true of variations in the level of phosphorus. The low yield in the 40-0-40 treatment is associated with a late-maturing condition of bolls on plants with low phosphorus. Whether there was actually a difference in total numbers of bolls eventually harvestable per plant within the phosphorus treatment is not known. A low level of potassium (40-80-0) seemed to result in a somewhat increased "total calcium" and calcium content, as did also a deficiency of all three elements ("no fertilizer"). It should be noted that the "40-80-0" and "no fertilizer" plots produced very small plants, with extremely low yields, also that from the arealometer values it is known that the fiber from these plants was so thin-walled as to be outside of the micronaire range normally acceptable in commercial fiber. The yields for these two plots (Table 7) were considerably below practical commercial levels, so that the effect of the fertilizer treatments on the calcium content of the fiber may have questionable relevance to the properties of current U.S. commercial cotton.

Relation to preharvest weathering

Between the time when the bolls open and the time when the fiber is harvested, cotton may remain in the field for several weeks. What influence does the weathering which occurs during this period have on the calcium and magnesium content of the fiber? Cotton bolls that had been harvested within a day or two of

TABLE 7.--Ash, "total calcium", calcium, magnesium, calcium-magnesium ratio, and yield of cotton fiber from plants grown under different fertilization regimes on the Keitt Plots, Florence, South Carolina, crop of 1960, (Note underlined values).

Treatment (Pounds per acre of N,P, and K)	Percent ash	"Total calcium", p.p.m.	Calcium, p.p.m.	Mag- nesium, p.p.m.	Ca/ Mg ratio	Arealo- meter value	Yield in lbs. seed cotton per plot
Variations in nitrogen level:							
0-80-40....	1.33	1050	550	300	1.83	447	78.5
40-80-40...	1.25	1200	500	425	1.18	463	107.2
50-80-40...	1.27	1050	500	325	1.54	507	118.4
100-80-40..	1.25	1100	475	375	1.27	429	75.0
Variations in phosphorus level:							
40-0-40....	1.23	1125	500	425	1.18	438	32.8
40-60-40...	1.19	1100	525	350	1.50	510	107.6
40-80-40...	1.25	1200	500	425	1.50	463	107.2
40-160-40..	1.28	1100	500	375	1.33	494	102.0
Variations in potassium level:							
40-80-0...	1.37	<u>1500</u>	<u>750</u>	450	1.67	602	7.7
40-80-40..	1.25	<u>1200</u>	<u>500</u>	425	1.18	463	107.2
40-80-80..	1.29	1275	575	425	1.35	457	98.3
No fertilizer.	1.18	<u>1400</u>	<u>750</u>	400	1.87	568	3.8

opening at Florence, S. C., were analyzed in comparison with cotton from similar bolls which had been subjected to rain and other elements of weathering for 10 weeks. The data (Table 8) show that there was a small loss of these elements from the fiber during weathering. Magnesium was lost more readily than calcium. In 1952 the ash content of the fiber decreased by 46 percent, the calcium decreased only 10 percent, and the magnesium 32 percent. Other ash constituents appear to be more readily leached out of the fiber than is calcium.

Variation from boll to boll

Are the amounts of calcium and magnesium in cotton fiber highly variable from one boll to the next on the same plant? In order to obtain data pertinent to this question, 11 bolls that had been picked with a minimum of weathering from a single plant of the variety Coker 100, at Florence, S. C., in 1960 were analyzed. The bolls were numbered from 1 to 11 in the approximate order of flowering from the earlier to the later bolls. The data (Table 9) indicate that neither

TABLE 8.--Results of determinations of ash, calcium, and magnesium on cotton fiber weathered in the field at Florence, S. C.

Sample (year of production and degree of weathering)	Rainfall (inches)	Percent ash	Parts per million		
			"Total calcium"	Calcium	Magnesium
1951, unweathered.....	--	1.32	1600	675	560
1951, weathered 10 weeks.....	5.52	1.07	1425	700	440
1952, unweathered.....	--	1.25	1300	525	470
1952, weathered 10 weeks.....	1.47	0.67	1000	475	320
1953, unweathered.....	--	1.27	1550	650	550
1953, weathered 10 weeks.....	4.26	1.06	1450	600	520
1954, unweathered.....	--	1.31	1475	650	500
1954, weathered 10 weeks.....	6.84	1.41	1375	650	440

TABLE 9.--Percent ash, "total calcium", calcium, and magnesium content for cotton fiber from eleven bolls taken from a single plant of the variety Coker 100, of the 1960 crop at Florence, S. C. (Bolls numbered according to approximate sequence of flowering.)

Boll number	Percent ash	Parts per million			Arealometer value
		"Total calcium"	Calcium	Magnesium	
1.....	1.40	1550	525	600	446
2.....	1.51	1550	550	575	509
3.....	1.40	1525	475	625	455
4.....	1.35	1550	500	625	448
5.....	1.41	1525	550	600	473
6.....	1.52	1550	500	625	477
7.....	1.38	1575	600	600	550
8.....	1.36	1550	550	600	433
9.....	1.33	1500	525	600	463
10.....	1.35	1500	550	575	477
11.....	1.41	1675	625	625	491
Average.....	1.40	1550	541	604	475

calcium content nor magnesium content exhibited a high degree of variation in fiber from one boll to the next. Further, for the plant analyzed, there was no clear indication of a trend in the magnitude of values from the earlier to the later bolls. Similar conclusions were obtained from analyses of fiber from a greenhouse-grown plant (Table 10). It may be noted that the calcium-magnesium ratio was much higher in the greenhouse plant (1.62) than in the Florence plant (0.90).

TABLE 10.--Percent ash, "total calcium," calcium, and magnesium content for fiber from twelve bolls from a single plant grown in the greenhouse

Boll number	Percent ash	Parts per million			Arealometer value
		"Total calcium"	Calcium	Magnesium	
1.....	1.03	1450	700	450	439
2.....	1.13	1400	650	450	467
3.....	1.10	1400	700	430	463
4.....	1.18	1550	650	550	465
5.....	1.15	1600	800	480	469
6.....	1.16	1450	700	450	565
7.....	1.11	1400	650	450	513
8.....	0.97	1450	800	400	530
9.....	1.04	1550	800	450	535
10.....	1.12	1500	750	450	529
11.....	0.99	1650	850	480	610
12.....	1.06	1700	900	480	595
Average.....	1.09	1508	746	460	515

DISCUSSION

Dyers concerned with the pronounced effects of calcium and magnesium on the shade of certain dyes applied to cotton have wondered whether the calcium and magnesium content of the fiber might be related to some area of growth or some growth condition. Although in a general way Middling Texas-Oklahoma and Western cotton tends to be somewhat higher in calcium and total calcium than Middling cotton from the Southeast and Midsouth, there is considerable variation in these properties from one specific location to another within each of these major areas and much overlapping of results between the two areas. Even though numerous cultural practices with cotton have changed in recent years, there seems to be no indication from the presently available data that such changes have resulted in any general increase in the calcium and magnesium levels in the fiber of U.S. cotton. The overall content of these elements in a sample of commercial cotton does appear to be influenced by the amount of leaf trash present.

Representatives of one prominent concern engaged in the marketing of raw cotton have called attention to the amount of foreign mineral material from the soil which seems to be present in some of the fiber supplied to them by producers. In this connection it might be well to observe that a high content of calcium and magnesium in commercial fiber could result from leaf fragments as well as from soil contamination. Duststorms are a common occurrence in various cotton-growing areas, and direct soil contamination of the lint, either during such storms or as a result of splashing of soil onto the lower bolls during rainstorms, undoubtedly does lead to increased mineral content of commercial fiber in some cases.

METHODS

The method of analysis used was adapted from that described by Patton and Reeder,³ and was carried out as shown below:

Materials and reagents

Total calcium and magnesium indicator: Dissolve 0.5 g. of Eriochrome Black T (F-241) in 60 ml of water with a drop of 6.5 N KOH plus 40 ml. of ethyl alcohol. Store in an amber dropping bottle in refrigerator.

Calcium indicator: Dissolve 0.0200 g. of 2 hydroxy-1- (2-hydroxy-4-sulfo-1-naphthylazo) -3-naphthoic acid in 50 ml. of distilled water and then add 50 ml. of alcohol with a drop of 6.5 N KOH. Store in amber bottle in refrigerator.

Standard disodium dihydrogen (ethylenedinitrilo) tetraacetate (analytical grade): Dissolve 2 g. of this reagent in exactly 1 liter of water. Standardize against the calcium chloride solution, using the Eriochrome Black T as the indicator and adjust to 0.1000 N by dilution.

Standard magnesium chloride: Dissolve 1.016 g. of reagent grade magnesium chloride hexahydrate in 1 liter of distilled water.

Standard calcium chloride: Carefully transfer 0.5004 g. of pure calcium carbonate (low in alkalies and heavy metals) to a 1-liter volumetric flask. Add 125 ml. of 0.1000 N hydrochloric acid and swirl the contents until solution is complete. Add 40 ml. of 0.100 N sodium hydroxide, cool and dilute to mark.

Buffer: Add 55 ml. of concentrated hydrochloric acid to 400 ml. of distilled water and mix thoroughly. Slowly add 310 ml. of redistilled monoethanolamine with stirring. A mixture of 100 ml. of standard magnesium chloride and 100 ml. of standard ethylenedinitrilo tetraacetate (the magnesium should be exactly sequestered) is added and mixed well. Dilute the mixture to 1 liter.

Potassium hydroxide: 6.5 N, reagent grade.

Preparation of samples

Ash a 2-gram sample of cotton at 550 C for at least 2 hours or until white. Carefully add 5 ml. of 1 N HCl to ash in crucibles. Let stand on steamtable a few minutes to dissolve; then rinse into a 50 ml. volumetric flask and dilute to the mark.

³Patton, J. and Reeder, W. 1956. New indicator for titration of calcium with (ethylenedinitrilo) tetraacetate. Anal. Chem. 28 (6):1026-1028.

Analytical procedures

Total calcium and magnesium: Pipette a 10 ml. aliquot of the prepared sample into a 50 ml. Erlenmeyer flask. Add 1 ml. of buffer solution and mix well. Add 2 to 4 drops of Eriochrome Black indicator and titrate to the blue end point or the point at which the red completely disappears with standard (ethylenedinitrilo) tetraacetate solution. Record the volume as the amount of the standard solution required for total calcium and magnesium.

Calcium: Pipette a 10 ml. aliquot of the prepared sample into a 50 ml. Erlenmeyer flask, add 1 ml. of 6.5 N potassium hydroxide, and mix. Allow the flask to stand for 3 to 5 minutes. Add 4 drops of calcium indicator and titrate with standard ethylenedinitrilo tetraacetate solution to the blue end point. A blank titration, replacing the sample with distilled water, is then made. One ml. of 0.010 N ethylenedinitrilo tetraacetate solution is equivalent to 0.2004 mg. of calcium.

Magnesium: Subtract the volume of standard tetraacetate required for the calcium from the volume required for the total calcium and magnesium on equal aliquots. One ml. of 0.010 N tetraacetate is equivalent to 0.1216 mg. of magnesium.

Even though measures were taken to eliminate ion interference in the total calcium titration using Eriochrome Black T, difficulties were still present. In some cotton ash samples, more indicator was needed near the end point to compensate for fading interference. When enough indicator was added, a purplish-blue end point was obtained, which remained stable for a few minutes. At this stable end point, 800 p.p.m. of added magnesium or calcium could be recovered. In most of the calcium determinations the end point was readily obtained.

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